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RESEARCH ON MARTENSITIC TRANSFORMATIONS IN IRON ALLOYS.(U)
NOV 77 C M WAYMAN

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20. ABSTRACT (Continue on reverse side if necessary and identify by block number) Ordering of nearly stoichiometric Fe-25 at.% Pt alloys causes a progressive change in the martensitic transformation from a nonthermoelastic to a thermoelastic type. Accompanying this are several pronounced property changes such as the development of martensite tetragonality and the shape memory effect, reduction of transformation hysteresis, morphology changes and a different mode of reverse transformation, a reduction in the martensite shape strain and driving force at the martensite start temperature,		

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and the onset of invar behavior. These various effects are interrelated. In order to obtain a thermoelastic transformation in Fe-Pt alloys, the martensite start temperature must be lower than a certain value.

Electron microscopy studies of various alloys have shown a highly variable substructure in thin foil martensites, compared to those formed in the bulk condition. In Fe-Ni and Fe-Ni-C alloys, the transformation twin width decreases with an increase in carbon content. This is believed to result from austenite strengthening. Similarly, a refinement of twin structure occurs after ausforming the parent phase prior to martensite formation. Substantial changes in the martensite substructure can also be brought about by ausaging to result in fine precipitates which impede transformation twin formation. In certain alloys, extra diffraction spots are observed in freshly formed martensite. These are attributed to a special kind of carbon atom ordering which disappears when the virgin martensite is heated.

Preliminary experiments have been carried out on pretransformation phenomena in several ferrous alloys.

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RESEARCH ON MARTENSITIC TRANSFORMATIONS IN IRON ALLOYS

Final Report

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U. S. Army Research Office

Grants DAHCO4 74 G 0134; 76 G 0176
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RESEARCH ON MARTENSITIC TRANSFORMATIONS IN IRON ALLOYS

I. Foreward

The present grant was initiated in March 1974, and has been concerned with electron microscopy studies of ferrous martensites, and austenite ordering and related consequences in Fe-Pt alloys. Structure-property-morphology relationships have been sought, and the effect of austenite ordering on transformation kinetics, thermoelastic behavior, the shape memory effect and related phenomena in Fe-Pt alloys has been studied. The work has proceeded as follows:

1. Electron Microscopy
 - A. Thin Foil Martensitic Transformations
 - A1. Extra Diffraction Spots Appearing in Freshly Formed Martensites
 - A2. Comparative Studies of the Substructure of Martensite Formed in Thin Foils
 - A3. Pretransformation Phenomena
 - B. Effect of Austenite Conditions on Martensite Substructure
 - B1. Twin Width Variation
 - B2. Effect of Ausforming
 - B3. Effect of Ausaging (Precipitation)
2. Austenite Ordering and Consequences in Fe-Pt Alloys
 - A. M_s Temperature
 - B. Morphology
 - C. Transformation Volume Change
 - D. Ordering Induced Tetragonality
 - E. Martensite Shape Strain Magnitude
 - F. Driving Force at M_s
 - G. Invar Behavior
 - H. Transformation Hysteresis
 - I. Mode of Reverse Transformation
 - J. Extent of the Shape Memory Effect and Morphological Studies

Some of the above work has by now been published. Highlights from the above research are described below.

II. Research Findings

A1. Extra Diffraction Spots in Freshly Formed Martensite

Thin foil martensites in Fe-8Cr-1C, Fe-6Mn-1C and Fe-3Mn-3Cr-1C (M_s below room temperature in all cases) have been observed after cooling prethinned austenitic foils in an electron microscope cold stage. In the as-transformed condition, the martensite in these materials shows extra superlattice reflections

which disappear when the martensite is heated to room temperature, and they do not reappear upon subsequent cooling. All the above alloys when in the bulk condition transform into martensite with a {225} habit plane. On the other hand, parallel experiments using Fe-Ni-C (0.2-0.7%C) alloys which form {3 10 15} martensite in the bulk do not show the extra spots.

A detailed analysis of the diffraction patterns exhibiting the extra spots shows a $1/6$ (110) periodicity. This has been interpreted to mean that carbon atoms fall in the wake of Frank type interface dislocations (1) which glide on {110} planes in the martensite (i.e., the inhomogeneous shear of the crystallographic theory). Such "sheets" of carbon atoms may act as a diffraction grating and produce the $1/6$ spots. During heating, these planar atmospheres are expected to be dispersed in the martensite lattice. (2)

A2. Comparative Studies of Thin Foil Martensites

Although it might be expected that some simplification in the substructure of martensite formed in thin foils would occur because certain bulk constraints are absent, this has been found not to be the case. In {225} type steels, {112} twins are observed, and in addition {110} and {123} stacking faults are found in the thin foil martensites. In {3 10 15} Fe-Ni-C and Fe-Ni alloys, both {112} twins and dislocations lying on {112} are observed in the thin foil martensite, as with bulk martensite. Generally, the martensite substructure is more variable and complex in the thin foil martensites, and further, the adherence to a strict habit plane is relaxed in thin foils. Curved habit "planes" are frequently observed and thus the typical morphologies and substructures of thin foil martensites are more difficult to characterize than for the bulk case. However, contrary to an earlier report (3) the orientation relationship after thin foil transformation is no different than that found in the bulk.

A3. Pretransformation Phenomena

Prethinned austenitic foils of Fe-8Cr-1C, Fe-6Mn-1C and Fe-30Ni alloys have been cooled in an electron microscope cold stage, and a lattice oscillation or "streaming effect" has been observed. (4) All these alloys have a subzero M_s temperature; the first two form {225} martensite and the last forms {3 10 15} martensite when transformed in the bulk. The oscillations appear to indicate a pretransformation phenomenon since their intensity increases as the M_s temperature is approached. Correspondingly, the intensity of diffuse streaking in electron diffraction patterns increases with decreasing temperature. The detectability of the streaming effect is very orientation dependent, being most obvious in {100} and {110} orientations and highly sensitive to the Bragg condition. In most cases, as the M_s temperature is approached localized regions will transform to martensite outside the field of view, and the area of interest and which is being observed is buckled out of contrast because of the transformation shear. Although clearly detectable, the results obtained to date are qualitative rather than quantitative (i.e., intensity vs. temperature) and further efforts are required.

B. Effect of Austenite Condition on the Martensite Substructure

B1. Twin Width Variation in Fe-Ni and Fe-Ni-C Martensites

Fe-33Ni, Fe-31Ni-0.23C, Fe-30Ni-0.39C and Fe-26Ni-0.72C alloys with subzero (and nearly equal) M_s temperatures and {3 10 15} habit martensites were transformed in the bulk and electrothinned for observations in the electron microscope. It was found that the {112} transformation twin width decreases with an increase in carbon content, from 102 Å at 0.23%C to 57 Å at 0.72%C, in the Fe-Ni-C "thin plate" martensite, which is characterized by twins of uniform width which extend completely across the plates from one interface to the other. The scatter in twin width measurements is less at the higher carbon contents (harder austenite), but in all cases observed the twin-matrix ratio remained unchanged, as expected from the crystallographic theory (i.e., constant habit plane). In the carbonless Fe-Ni alloy, the transformation twins taper away from the midrib region and the twin density becomes lower. However, the twin width at the midrib, 140 Å, is that expected by extrapolating the results for the Fe-Ni-C alloys to 0%C, indicating the austenite hardening (by carbon solid solution strengthening) causes a substantial twin refinement.

An unusual austenite dislocation structure consisting of a high density of looped dislocations was found adjacent to martensite plates in the Fe-Ni-C alloys. These dislocations are believed to be formed by a reverse transformation (backwards interface movement) during heating the martensite from -196°C to room temperature, and are much less prevalent in the higher carbon alloys where the transformation twin spacing is smallest. These dislocations indicate that "thin plate" martensites are somewhat reversible, and similar observations have been made for partially ordered Fe-Pt alloys.(5)

B2. Effect of Ausforming on the Martensite Substructure

Partially twinned Fe-33Ni martensites ($M_s = -105^\circ\text{C}$) and completely twinned Fe-31Ni-0.23C martensites ($M_s = -170^\circ\text{C}$) were studied after room temperature ausforming from 0 to 70% reduction by multipass rolling. For both alloys ausforming produces a dislocation cell structure in the austenite which is inherited without modification, by the martensite. In the Fe-Ni alloy the cell structure is found in both the twinned (midrib) and untwinned (interface) regions. The untwinned regions of such plates contain a regular (transformation) network of dislocations, which remains unaltered by the austenite cell structure. Similarly, in the Fe-Ni-C alloys, the transformation twins are unimpeded by the prior cell structure. Since no carbide precipitation occurred under the ausforming conditions employed, it is clear that carbide precipitation during ausforming is not necessary to "pin" the cell structure. It is also clear that the martensite-austenite interface, backed by either dislocations or twins, does not sweep up the dislocations induced by ausforming.

However, even though the martensite twins are uninhibited by the austenite cell structure, they do undergo a refinement with increased degrees of ausforming. For the Fe-31Ni-0.23C alloy, the twin spacing is 53 Å after 70% ausforming, compared to 102 Å for 0% ausforming. Since the twin spacing decreases with the amount of ausforming and an increase in carbon content, it is indicated that the martensite twin size depends on the austenite hardness.

B3. Effect of Austenite Precipitates on the Martensite Substructure

The influence of coherent, spherical, Cu_3Au type γ' precipitate particles formed by ausaging on the martensite morphology and substructure in an Fe-31Ni-4Ti-0.2C alloy has been studied. In this alloy, lenticular martensite plates form. By varying the aging time at a given temperature (720°C) both "fine" (50 Å) and "coarse" (250 Å) evenly spaced precipitations were obtained, resulting in the same austenite hardness and subzero M_s temperature. The martensite morphology was unaffected over the range of precipitate sizes studied, and the plates were typically lenticular with a midrib region, even in the unaged condition. However, despite no change in morphology, the martensite plate substructure was highly sensitive to the size of the γ' particles. Precipitates in the 50 Å size range impeded twin formation; the transformation twins became highly segmented and irregular, but there was no change in the extent of the twinned volume in the plates, comparing aged and unaged specimens. But for the coarse γ' precipitates the transformation twins were suppressed and only a few scattered twins along the midrib region were observed.

The γ' particles are fcc in the austenite and remained so during transformation to martensite, thus ending up in an incoherent state with respect to the martensite. However, these particles are rotated during transformation, to conform with the martensite lattice rotation relative to the austenite.

2. Austenite Ordering and Consequences in Fe-Pt Alloys

Fe-Pt alloys containing 23, 24 and 25 at.% Pt have been studied. As reported earlier, (6) austenite ordering leads progressively to a change from a non-thermoelastic, burst type transformation (as in Fe-Ni alloys) to a thermoelastic transformation (as in Cu-Zn alloys). In the well ordered highly thermoelastic alloys the shape memory effect has been observed; specimens deformed below M_s to as much as 9% strain will recover their shape when heated to the A_f temperature. Our previous work showed no obvious change in the transformation crystallography following an ordering treatment, and the reason(s) for the marked difference in kinetic behavior in the ordered alloys has been sought. Detailed studies of a number of properties as a function of ordering time (degree of order) have been made, and it has been determined that increased austenite ordering results in:

- a) A lowering of the M_s temperature. The M_s temperature in a disordered Fe-25 at.% Pt alloy is 20°C, while that for the same alloy ordered 100 hours at 650°C is below -196°C.
- b) A morphology change. In the disordered alloys, classical lenticular martensite plates are formed, but in the ordered alloys which show thermoelastic behavior, the plates are of the "thin plate" type, with very parallel interfaces. Moreover, the thin plates form in a very systematic and cooperative manner, without bursting and the end result is that quite large specimen volumes contain only four (of 24 possible) habit plane variants. This is in contrast to the disordered alloys where many autocatalytically formed variants comprise a given region. It appears that the strain energy is dissipated for the lenticular plates and stored for the thin, thermoelastic plates.

- c) The transformation volume change is lowered. For example, $\Delta V = 1.4\%$ for a disordered alloy and 0.5% when the same alloy is well ordered.
- d) The martensite becomes tetragonal rather than cubic ($c/a = 1.12$ for ordered Fe-25 at.% Pt).
- e) The shape strain magnitude is reduced from 0.21 to 0.16 as a consequence of the ordering induced tetragonality.
- f) The driving force at M_s is reduced from an estimated 300 to 5 cal/mole, and measurements of the transformation enthalpy change show ΔH is reduced from 570 to 80 cal/mole upon ordering.
- g) Invar behavior sets in because the austenite Curie temperature is raised above M_s during ordering (and hence the small transformation volume change).
- h) Decreased transformation hysteresis. Cycling of ordered martensites produces disorder, raises the hysteresis, and establishes a "micro-structural memory" because of the repeated enlistment action of select nuclei induced by the cycling disorder.
- i) Change in the mode of the reverse transformation. The well ordered plates reverse by simply shrinking or the nucleation of only a single variant of the parent phase within each plate. In disordered alloys, many orientations of the parent are nucleated in each martensite plate.
- j) An increase in the extent of the shape memory. Strains as high as 9% are recovered in a well ordered alloy, whereas disordered alloys show no shape memory.

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